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COMPLETE SPECIFICATION

Improvements in or Relating to Distillation

We, VICTOR WOLF LIMITED, a British Company of Victoria Works, Croft Street, Clayton, Manchester, 11, in the County of Lancaster, do hereby declare the nature of this invention which has been communicated to us by Dr. HANS-BERNHARD SEEBOHM, a German National, of Steinau, Krs. Schluechtern, Germany, and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention is concerned with improvements in or relating to distillation, and is more particularly concerned with a process and an apparatus for the condensation of the vapours produced during distillation.

Hitherto, the condensation of these vapours has been effected, for example, in large empty cylinders on the interior walls of which the vapours are entirely or partially condensed on account of the cooling effect of the air which flows around the cylinders. One can cut out any desired fractions from the vapours by this method without using any special device, by using several subsequent cylinders and by collecting the condensate fractions separately. This device has the advantage of simplicity, but it also has several concomitant disadvantages. Firstly, it is not possible to effect condensation at an exact temperature, because of the movement of the surrounding air and because the temperature of this air is always varying, with consequent variation in the coefficient of heat transmission from the outer surface of the cylinder to the air and accordingly the amount and the composition of the condensate produced in any one cylinder also varies quite considerably.

In addition, the coefficient of heat transmission from the outer surface of the cylinder to the air is small and this necessitates the building of large cylinders in order to get an adequate cooling effect. Further, the total heat of evaporation is lost by transmission to the surrounding air.

More difficult fractional condensations are usually carried out in very high columns provided with packing material, bubble caps or sieve plates. Such pieces of apparatus in spite of being very expensive, are used everywhere in the Chemical Industry, especially in the refining of petroleum. Although the fractionating effect of such apparatus is good, considerable resistance is offered to the vapours and this is particularly troublesome when distilling under reduced pressure. Moreover, the heat of evaporation is lost by being transferred into the reflux condenser.

A still head is known which comprises one or more continuous helical planes wound round a core. This core may be hollow and be supplied with a current of a liquid, air or the vapour of a suitable liquid in order to regulate the temperature of the apparatus; the fluid in the core flowing in a direction parallel to that of the fluid flowing in the helical planes. This still head is obviously suitable only for laboratory use and is of no value in large scale practice since it can only be enlarged lengthwise and this would cause undesirable pressure in the vapour space.

All these disadvantages can be overcome by utilising one or more features of the present invention. Thus for example in the present invention the vapours to be cooled flow at right angles to the cooling fluid and it is possible to enlarge the apparatus by installing side by side any number of pipes fitted with fins, so overcoming the disadvantages of the last-mentioned still head.

According to the present invention, a process of distilling and condensing is provided which comprises disposing at least one calorifier, as herein defined, and on the outer surface of which the vapours are condensed in the vapour outlet from the still.

The invention also includes a distillation apparatus comprising a still, means for heating the material in the still and a vapour outlet from the still in which at least one calorifier, as herein defined, is disposed in the vapour outlet from the still.

Whenever the term "calorifier" is used throughout the specification it means a heat exchange device having pipes through which

cooling fluid is circulated and in which, by providing the pipes with fins, the area of the surface, across which the heat is extracted from the vapour to be cooled, is substantially greater than the area of the surface across which the heat is transferred to the cooling fluid in the pipes; the vapours to be cooled flowing in a direction at right angles to the cooling fluid.

In the present invention, due regard is paid to the fact that the coefficient of heat transmission between the vapour to be cooled and the heat exchange device on the one hand, and the heat exchange device and the cooling fluid on the other hand, may be different.

Such heat exchange devices are well known of course for pre-heating air with steam and for cooling air with cold water, the gas flowing round the fins, but so far, however, such devices have not been used in the technique of distillation and condensation.

In carrying out the process of this invention, the pipes of the calorifier are filled with any suitable cooling fluid—which in most cases will be water—whilst the vapours to be condensed are flowing around the considerably enlarged outer surface. Boiling liquids, for example, water or any suitable organic liquid, under any desired pressure, may be used to feed the calorifiers and by this very simple means condensation at an exact temperature is made possible.

The vapours of the cooling liquid which are generated can be utilised in any suitable way and thereby the heat used in distillation can at least partially be recovered. This heat can at least partially be recovered also when using liquids which are not boiling, but the advantages to be gained are not so great.

The liquid in the cooling pipes of the calorifier can be made to boil under such a pressure that the desired difference of temperature is effected which corresponds to the amount of condensation calculated and desired. Very often this vapour pressure is so high that the vapours produced can be used, for example, for heating and drying purposes or by introducing them into the product to be distilled, and a considerable part of the amount of the heat spent in the distillation is thus recovered.

The fins of the calorifier present a large surface of entirely uniform temperature effected by the quick heat transfer, and this surface is many times larger than that of the pipes on which the fins are fitted, and can vary considerably. The amount of condensate formed on this surface depends on the coefficient of overall heat passage of the calorifier and on the difference in temperature between the vapours to be condensed and the cooling fluid circulating or evaporating in the pipes. By keeping the temperature of this cooling fluid constant, for instance, by keeping the pressure of the boiling liquid constant, it is possible to keep constant the quantity and composition of any particular fraction of the condensate. Each fin, therefore, is used not only for heat exchange, but also as a surface of exact temperature, on which surface a process such as condensation or rectification can take place.

The calorifier can be used in several different positions. If several calorifiers are placed in super-position, and if the vapours are passed over them from the top to the bottom, a progressive condensation takes place. If the vapours pass over these calorifiers from the bottom to the top, fractionation takes place on the large surface of the fins on which condensate meets the rising vapours. If the calorifiers are placed in a slanting position, the fins act like cascades, whilst the circulation of the cooling liquid inside the pipes is favourably affected. The calculated amount of the vapours is also condensed when a horizontal current of vapours passes through the calorifiers placed horizontally one after the other.

There is no limit to the number of calorifiers to be used; it simply depends on the material to be distilled and the purpose of the distillation. The disposition of the calorifiers, namely whether they are to be placed one after another horizontally or one on top of another, and the choice of the cooling fluid to be used, also depend on the same factors. By using suitable cooling liquids with appropriate boiling points it is possible to effect an easy and exact vapour fractionation, and this is of great importance in many branches of industry, for example, in the distillation of mineral oils, tars and fatty acids.

In order to achieve a still better fractionation, it is also possible to use the calorifiers at the same time as condensers and evaporators. This can be effected by providing the calorifiers with two pipelines, the upper one of which is fed by a liquid whose temperature is lower than that of the liquid film flowing across the finned tubes, and the lower one of which is fed by a liquid whose temperature is higher than that of this liquid film.

As can be seen from the above, the use of calorifiers according to this invention in the technique of distillation and condensation has considerable advantages. The cooling effect is many times increased in comparison with such apparatus as has been used hitherto and yet the free escape of the vapours produced during distillation is not hindered; the heat consumed by distillation may be recovered to a larger extent; easy and exact fractionation of the vapours gener-

ated during distillation is made possible, and the relatively simple plant requires very little space. The invention possesses a very wide scope and is applicable to the condensation of organic or inorganic substances.

The invention will now be further illustrated by reference to the accompanying drawing, which is purely illustrative, and in which:—

Figure 1 is a diagrammatic representation of a still with three calorifiers fitted horizontally one after the other, and

Figure 2 is a diagrammatic representation of a still with five calorifiers fitted one on top of the other.

In the apparatus shown in figure 1 which, for instance, is suitable for the distillation of crude glycerine, the still 11 is filled through supply pipe 12 with crude glycerine which is distilled at 20 mm. mercury (abs.) by means of coil 13 heated by steam at 16 atmospheres gauge pressure. The glycerine vapours escape through duct 14 where three calorifiers—15, 16 and 17—with horizontally fitted fins are placed one after the other. In the pipes which carry the fins of the calorifier 15, water is boiling at 4 atmospheres gauge pressure (151° C.). The steam which is generated rises through pipe 18 into a vessel 19, where it is condensed, and the water returns through pipe 20 to the calorifier 15. Steam, preferably superheated, passes from vessel 19 through pipe 21 into the still 11, in support of the distillation process. This steam may, of course, also be used in any other suitable way. Similarly, the calorifier 16, is connected through a pipe 18, with a vessel 19, from which the water is returned to the calorifier through a pipe 20 and from which the steam can also be taken by passing through a pipe 21 and be used in any suitable way. The calorifier 17 which serves to condense the residual vapours is fed through supply line 22, with cold water, which is discharged through pipe line 23. The glycerine which condenses on the fins of the calorifiers is discharged in the direction of the current of vapour and is drawn off through pipes 24.

Make-up cooling water for the calorifiers 15 and 16 may be introduced through pipe lines 34.

The cooling surface of the pipes may be 5%, and the surface presented to the vapour being cooled may be 25%, of the surface of the air-cooled cylinders used up to now. The space necessary for this apparatus is even smaller in view of the construction of the calorifiers. The quantity of steam at 4 atmospheres gauge pressure is more than half that of the steam at 16 atmospheres gauge pressure and as already mentioned, it can, after superheating, be introduced into the boiling crude glycerine, or can be passed into a supply line for steam at 4 atmosphere gauge pressure.

In Figure 2, where parts which correspond to those in Figure 1 have the appropriate reference numbers, the high vacuum still 25 of known construction, from which a fatty acid mixture is distilled at 5 mm mercury (abs.) is heated in a suitable manner. The vapours enter a column 26 from the bottom which column contains five calorifiers 27, 28, 29, 30 and 31, arranged one on top of another. Between each pair of calorifiers there is a baffle plate 32, from which the condensate of the calorifier arranged on top of it is discharged. The two lower calorifiers 27 and 28 are cooled with a boiling mixture of 50% diphenyl and 50% diphenyloxide, whilst the upper ones 29, 30 and 31 are cooled with boiling water. The vapours of the fatty acids are condensed fractionally on the fins at an exactly defined temperature, so that a practically pure fatty acid is obtained in each calorifier. The variations in the mixture of crude fatty acids can be taken into account by varying the pressure of the liquids in the pipes of the various calorifiers so that only such an amount of the fatty acid vapours is condensed as corresponds to the portion of one fatty acid in the vapour mixture. The cooling liquids are circulated by the pumps 33 from vessels 19 and pipe lines 20, through the cooling pipes of the calorifiers, as is the practice in known types of positive circulation evaporators, in order to increase their capacity of heat absorption. The vapours of the cooling liquids return through pipelines 18 to the vessels 19. From here, they can be taken through pipe-lines 21 and suitably utilised and then be returned by pipelines 34. The condensate which is discharged from the various baffle plates 32 through pipe-lines 24 can be poured (entirely or partly) over the respective lower calorifiers in order to achieve a better fractionation or it can be returned to still 25.

In both these plants, the pressure at which the cooling medium boils may be controlled by valves (not shown) on pipe 21. The process of this invention can be carried out in a continuous or discontinuous manner and steam can be used as carrier for the distillate.

Having now particularly described and ascertained the nature of the said invention and in what manner the same is to be performed, as communicated to us by our Foreign correspondent, we declare that what we claim is:—

1. Process of distilling and condensing which comprises disposing at least one calorifier, as herein defined, and on the outer surface of which the vapours are condensed, in the vapour outlet from the still.

2. Process as claimed in claim 1 in which a boiling liquid is used as the cooling me-

dium for the calorifier.

3. Process as claimed in claim 2 in which the boiling liquid is water, under super-atmospheric pressure.

4. Process as claimed in any of claims 1, 2 or 3 in which a plurality of calorifiers is used and each subsequent calorifier is maintained at a lower temperature than the one which is immediately nearer to the still, thus effecting fractional condensation.

5. Process as claimed in any of claims 1 to 4 in which at least part of the heat content of the vapour of the cooling liquid in the calorifier is utilised in the distillation, for example, by introducing the vapour into the material to be distilled.

6. Process as claimed in any of claims 1 to 5 in which a plurality of calorifiers are disposed either in superposition or horizontally one after the other.

7. Process as claimed in any of claims 1 to 6 in which a different liquid is fed into the upper portion of the calorifier from that which is fed into the lower portion of the calorifier.

8. Distillation apparatus, comprising a still, means for heating the material in the still and a vapour outlet from the still, in which at least one calorifier, as herein defined, is disposed in the vapour outlet from the still.

9. Distillation apparatus as claimed in claim 8 in which means are provided for the utilisation in the distillation of at least part of the heat content of the vapours of the cooling liquid in the calorifier.

10. Distillation apparatus as claimed in claim 8 or 9 in which a plurality of calorifiers are disposed in superposition.

11. Distillation apparatus as claimed in claim 8 or 9 in which a plurality of calorifiers are horizontally disposed.

12. Distillation apparatus as claimed in any of the preceding claims in which the upper portion of the calorifier is made to act as a condenser and the lower portion made to act as an evaporator by feeding different liquids into the upper and lower portions of the calorifier.

13. Process of distilling and condensing as herein particularly described and ascertained.

14. Distillation apparatus substantially as herein described with reference to and as illustrated in Figure 1 or Figure 2 of the accompanying drawing.

Dated this 20th day of April, 1949.

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Chartered Patent Agents.

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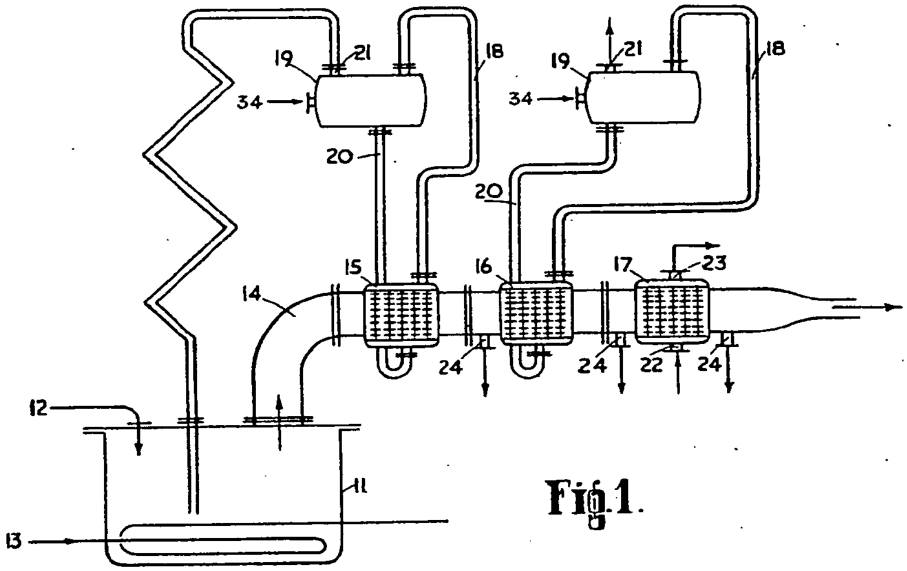

Fig.1.

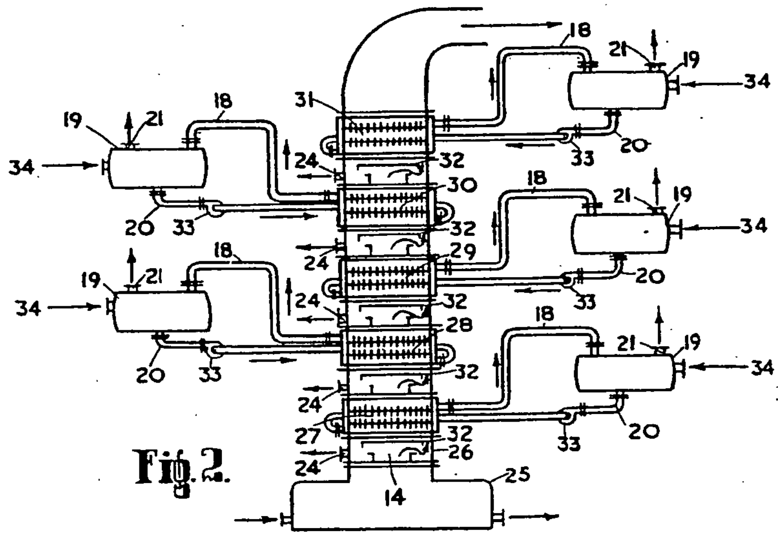

Fig.2.

H.M.S.O. (M.F.P.)